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1. Doc... US 6495449 B1

L7: Entry ... File: U... Dec 17, 2002

US-PAT-NO: 64...
DOCUMENT-IDEN...: US 6495449 B1

TITLE: Mult... diffusion barrier structure for improving adhesion pr...

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L5: Entry 3 of 4 File: USPT Nov 6, 2001

DOCUMENT-IDENTIFIER: US 6313035 B1
TITLE: Chemical vapor deposition using organometallic precursors

Brief Summary Text (10):
This invention in one respect, relates to a method of depositing a multi-component oxide layer on a semiconductor substrate by exposing the semiconductor substrate to gaseous organometallic precursor and a reactive gas under conditions effective to cause the gaseous organometallic precursor and reactive gas to combine and deposit a multi-component oxide layer on the semiconductor substrate.

Brief Summary Text (13):
In another respect, this invention relates to a method of depositing a layer of titanium silicon oxide on a semiconductor substrate. The method comprises the steps of positioning the semiconductor substrate within a semiconductor processing chamber, and introducing gaseous reactants including titanium organometallic precursor, reactive silane-based gas and gaseous oxidant into the semiconductor processing chamber under conditions effective to cause the gaseous reactants to deposit a layer of titanium silicon oxide on the semiconductor substrate.

Brief Summary Text (15):
In another respect, this invention relates to a method of depositing a multi-component layer comprising two or more nitrides on a semiconductor substrate by exposing the semiconductor substrate to gaseous organometallic precursor and reactive gas under conditions effective to cause the gaseous organometallic precursor and reactive gas to combine and deposit a multi-component layer on the semiconductor substrate.

Detailed Description Text (4):
Multi-component films also include multi-component oxide films. By "multi-component oxide film" it is meant that a film is comprised of at least one dielectric or conductive oxide compound in combination with another material. Examples of such multi-component oxide films include, but are not limited to, films containing more than one oxide compound or films containing a mixture of oxide and nitride compounds. These films may be deposited using more than one organometallic precursor, or by using an organometallic precursor and other reactive gases, such as a reactive silane-based gas and/or a gaseous oxidant. In the practice of this disclosed method, it is typical that deposition of these films occur at low temperatures and in the absence of plasma to activate deposition.

However, those skilled in the art will recognize that benefits of the disclosed method may also be obtained at higher process temperatures and by using plasma.

Detailed Description Text (6):
Although titanium silicon oxide is deposited in one embodiment, other multi-component oxide films may be deposited using different gaseous reactants. These other films include, but are not limited dielectric films such as tantalum silicon oxide, mixtures of titanium oxide and silicon nitride, mixtures of aluminum oxide and silicon oxide, mixtures of aluminum oxide, silicon oxide and titanium oxide and other combinations of these and other compounds. Also included are conductive films including a ruthenium oxide component, such as mixtures of titanium nitride and ruthenium oxide. During deposition, energy is supplied to a semiconductor substrate to cause a reactive deposition to take place, resulting in deposition of mixed phase metal silicon oxide onto the substrate surface. The process may be carried out in any semiconductor processing chamber or other environment known to the art that is suitable for depositing thin films on semiconductor substrates from organometallic precursors. For example, commercially available deposition equipment from suppliers such as Applied Materials, Novellus and/or Genus may be utilized. Particular models of such equipment include Applied Materials 5000D, Novellus Concept I and Genus 8700.

Detailed Description Text (8):
In one embodiment of the disclosed method, titanium organometallic precursors of the formula Ti(NR.sub.2).sub.4 may be used, where R is selected from the group consisting of H and a carbon containing radical, such as an alkyl. Specific examples of suitable titanium precursors include tetrakisdimethyl aminotitanium [Ti(N(CH.sub.3).sub.2).sub.4] (TDMAT) and tetrakisdiethylamino titanium [Ti(N(C.sub.2 H.sub.5).sub.2).sub.4] (TDEAT). Other suitable titanium precursors include bis (2,4-dimethyl-1,3-pentadienyl) titanium (BDPT), cyclopentadienylcycloheptatrienyltitanium (CpTiCht) and biscyclopentadienyltitanium diazide (Cp.sub.2 Ti(N.sub.3).sub.2). TDMAT is the typically used titanium precursor. Organometallic precursors, such as TDMAT, are typically liquid and may be vaporized for the CVD reaction. In other cases the organometallic precursor may be a solid which is sublimed to a vapor for reaction in the CVD chamber, such as bis[ethene 1,2(N,N'-dimethyl) diamide] titanium (IV), cyclooctatetraenetitanium, and tri(cyclooctatetraene) dititanium. In the practice of the disclosed method, a carrier gas, reactive gas or mixtures thereof may optionally be utilized to assist in vaporization and/or transportation of an organometallic precursor.

Detailed Description Text (9):
Because titanium organometallic precursors are used as a Ti source rather than titanium tetrachloride, the disclosed method offers the advantage of depositing titanium silicon oxide layers that are substantially free of chlorine related impurities when non-chlorine containing silicon reactive gases are used.

Detailed Description Text (12):

In the presen: :bodiment, the typical gaseous oxidant is O.sub.2. However, any r gaseous oxidant containing oxygen would be suitable, for .mple, ozone (O.sub.3), nitrous oxide (N.sub.2 O), nitrous dioxi: NO.sub.2), or high temperature mixtures of oxygen and water vap r oxygen and hydrogen. The carrier gas may be selected from noble or nonreactive inert gas, such as helium, argon or nitr ., or alternatively may be a reactant gas, such as a gaseous oxi: , hydrogen or other reactive gas. Typically the carrier gas i: gon.

Detailed Desc: ion Text (16):
Using the dis: ed method, deposition of titanium silicon oxide is typically car: out in the absence of plasma in a cold wall CVD reactor. Howev the method may also be practiced in other types of environmen: r semiconductor processing chambers, including, for example, non-plasma hot wall CVD process, a plasma cold wall or hot w CVD process, or in a radiant energy or rapid thermal CVD p: ss. When carried out in the absence of plasma, the method offers advantages of substantially eliminating the creation of h leakage damage centers in deposited dielectric layers and of orporating substantially no hydrogen into these deposited lay

CLAIMS:

6. The method claim 4, wherein the gaseous titanium organometalli ecursor comprises at least one of tetrakisdimet mino titanium (TDMAT), tetrakisdiethylamino titanium (TDE titanium ethoxide, titanium tetra-i-propoxide or a mixture the .

9. The method claim 8, wherein the gaseous titanium organometalli ecursor comprises at least one of tetrakisdimet mino titanium (TDMAT), tetrakisdiethylamino titanium (TDE titanium ethoxide, titanium tetra-i-propoxide or a mixture the .

14. The metho claim 8, wherein the step of exposing comprises the steps of:

positioning t emiconductor substrate within a semiconductor processing ch r; and

introducing aseous titanium organometallic precursor, reactive silane-based and gaseous oxidant into said semiconductor processing ch r.

15. The metho claim 8, wherein the step of exposing comprises the steps of:

positioning t emiconductor substrate within a semiconductor processing c r that is a non-plasma, cold wall, chemical vapor deposition (chamber;

pressurizing chamber; and

heating the s onductor substrate to a temperature, said pressure

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L7: Entry 1 of 1 File: USPT Dec 17, 2002

DOCUMENT-IDENTIFIER: US 6495449 B1
TITLE: Multilayered diffusion barrier structure for improving adhesion property

Abstract Text :
A method has been provided for improving the adhesion of copper to a nitrided metal diffusion barrier material, such as TiN, in an integrated circuit substrate. The method provided a multilayered diffusion barrier structure, comprising a nitrided metal diffusion barrier layer and an oxy-nitrided metal layer. The formation of an oxy-nitrided metal layer, instead of an oxide layer, permits the optimization of both contact resistance and adhesion property. The oxy-nitrided metal layer is formed either by the partial incorporation of oxygen into the nitrided metal diffusion barrier or by deposit[illegible] in an oxygen ambient.

Brief Summary Text (2):
This invention relates generally to integrated circuit processes and fabrication, and more particularly, to a method of improving the adhesion property of a diffusion barrier structure.

Brief Summary Text (9):
Various means have been suggested to deal with the problem of copper diffusion into integrated circuit materials. Several materials, including metals and metal alloys, have been suggested for use as barriers to prevent the copper diffusion process. The typical conductive diffusion barrier materials are TiN, TaN and WN. Addition of silicon into these materials, TiSiN, TaSiN, WSiN, could offer improve[illegible] in the diffusion barrier property. For non-conductive diffusion barrier, silicon nitride has been the best material so far. However, the adhesion of copper to these diffusion barrier materials has been, and continues to be, an IC process problem.

Brief Summary Text (11):
In a typical copper CVD process, copper is combined with a ligand, or organic compound, to make the copper volatile. That is, copper becomes an element in a compound, called precursor, that is vaporized into a gas. Selected surfaces of an integrated circuit, such as that of diffusion barrier materials, are exposed to the copper gas in an elevated temperature environment. When the copper gas compound decomposes, copper is left behind on the selected surfaces. Several copper precursors are available for use with the CVD process. It is generally accepted that the configuration of the

copper precursors, at least partially, affects the ability of the copper residue to adhere itself to the selected surfaces. Although certain precursors may improve the copper adhesion process, variations in the diffusion barrier surfaces to which the copper is applied, and variations in the copper precursors themselves, often result in unsatisfactory adhesion of copper to a selected surface.

Brief Summary Text (13):
It has become a standard practice in the semiconductor industry to apply CVD copper immediately after the deposition of the diffusion barrier material to the integrated circuit to improve the adhesion and to reduce the contact resistance. Typically, the processes are performed in a single chamber or an interconnected cluster chamber. It has generally been thought that the copper layer has the best chance of adhering to the diffusion barrier material when the diffusion barrier material surface is clean and free of contaminants. Hence, the diffusion barrier surface is often kept under vacuum, or in a controlled environment, and the copper is deposited on the diffusion barrier as quickly as possible. However, even when copper is immediately applied to the diffusion barrier surface, problems remain in keeping the copper properly adhered.

Brief Summary Text (14):
Charneski et al., U.S. Pat. No. 5,909,637, entitled "Copper adhesion to a diffusion barrier surface and method for same", proposed a method to use reactive gas species to clean the surface of the diffusion barrier to improve the adhesion to the subsequently deposited copper layer. This method has very limited success and often does not provide enough adhesion to be practical. Nguyen et al., U.S. Pat. No. 5,913,144, entitled "Oxidized diffusion barrier surface for the adherence of copper and method for same", further proposed a method to use reactive oxygen species to oxidize the diffusion barrier surface to improve the adhesion to the subsequently deposited copper layer. This method works well to improve the adhesion property, but by oxidizing the barrier material, it produces a non conductive layer that significantly increases the contact resistance of the integrated circuit even at a very small thickness.

Brief Summary Text (15):
It would be advantageous to understand the mechanism of the adhesion of CVD copper to a diffusion barrier material surface.

Brief Summary Text (16):
It would be advantageous to employ a method of improving the adhesion of CVD copper to a diffusion barrier material surface without oxidizing the diffusion barrier, without producing a non conductive layer and without increasing the contact resistance.

Brief Summary Text (17):
It would be advantageous to employ a method of improving the adhesion of CVD copper to a diffusion barrier material surface that can be optimized with respect to the contact resistance.

Brief Summary Text (18):
Accordingly, a method of improving the adhesion to the diffusion barrier surface is provided based on the analysis and understanding

of the proper s of the diffusion barriers. A good diffusion barrier, by d nition, does not allow foreign materials to diffuse through. It a has poor chemical bonding with many foreign materials. It not thermodynamically favorable for the diffusion barriers to with other materials because the resultant heat of formation is tive, that is, additional energies are required for these rea ons. Therefore the adhesion property of good diffusion bar materials based on chemical reaction is very poor. Deposit techniques that depend on chemical reactions to provide stron dhesion, such as chemical vapor deposition or electrochemic plating techniques, will be much more difficult to achieve good esion. In addition, copper material does not adhere well to many er materials, and thus adhesion of CVD copper to the diffusion rrier is even more difficult.

Brief Summary xt (19):
From thermody ic arguments, good diffusion barriers for copper thus are those hat have a positive heat of formation with copper. The nitrided al diffusion barriers such as TiN, TaN, WN, TiSiN, TaSiN, WSiN, posed of mainly nitrogen and other metallic components s as Ti, Ta, W, offer the best conductive diffusion barrier prop against copper diffusion. Copper nitrides have positive heat f formation so that it is thermodynamically unfavorable copper to bond to nitrogen. These nitrides are more thermodynami stable than copper nitrides and thus are effective ag copper bonding and diffusion. Of all the mentioned nit es, TiN and WN are similar to each other. TaN is a somewhat bett barrier than either TiN or WN, and thus adhesion of CVD copper to N is much more difficult to achieve than to TiN or WN. The adhe property of copper to an effective diffusion barrier how s poor.

Brief Summar xt (20 :
To improve t dhesion of the nitrided metal diffusion barrier to copper, it is perative to change the surface properties of the nitrided meta ffusion barrier layer. This surface should contain materials th nd with copper. Thermodynamically, it means that the compoun reactions of this material and copper should have a negative h f formation, such as a copper oxide. One way to change the s e properties of the nitrided metal diffusion barrier thus o form an oxy-nitride layer on the surface by oxy-nitridi nitrided meta diffusion barrier layer surface, and the pres of oxygen in the oxy-nitride layer would promote the adhesion copper to the diffusion barrier. The oxy-nitride layer compr he metal, nitrogen, and oxygen, and serves to improve the sion of the multilayered diffusion barrier structure to subsequently deposited layer. As oxygen is incorporated the nitrided metal diffusion barrier surface, the surface beco n oxy-nitrided metal, and no longer has the strong diffusion ba property. This surface thus exhibits strong chemical bon with the subsequent deposited layer. Furthermore, this thin ox rided layer is conductive and thus the contact resistance luced. The amount of oxygen in the oxy-nitride layer howeve a strong affect on the contact resistance. The oxidizing c on will produce an oxide layer, however thin, that raises the t resistance because of its non conductive property. Ca ould be taken to avoid the extreme case of

oxidizing th rided metal diffusion barrier that occurs by introducing cessive amount of oxygen. One way to avoid over-oxidizi e nitrided metal barrier surface is to introduce a small amount xy-nitride contained gas or a precursor that contains oxy nd nitrogen to the barrier surface. The introduction he oxy-nitride or oxygen-nitrogen combination reduces oxid of the surface. Therefore a method to improve the adhesion of itrided metal diffusion barrier without introducing -conductive oxide layer is to have a multilayer: a nitrided met ffusion barrier layer to block foreign material diffusion, a thin oxy nitrided metal layer to provide the adhesion pro . The oxygen content in this oxy-nitrided layer is small such t he nitrided metal layer is not oxidized and results in a conductive layer.

Brief Summar t (21):
In the oxy-n ing process, the metal comes from the nitrided metal diffus arrier layer, oxygen comes from the ambient, and nitrogen can rom the nitrided metal diffusion barrier layer and the ambient. e are different ways of incorporating the metal, oxygen, nit to form the oxy-nitride layer. One way is to introduce a amount of oxygen to a thin surface of the nitrided met ffusion barrier. To provide a thin oxy-nitrided metal layer as TiON, TiSiON, TaON, TaSiON, WON, WSiON, the nitrided met ffusion barrier surface is oxy-nitrided in an oxygen-conta ambient. The oxy-nitrided metal adhesion property improvement st effective with TiN, a less effective diffusion barrier, and less effective with TaN, a more effective diffusion ba . This method uses the nitrogen from the nitrided diffusion ba layer. Another way is to introduce oxygen and nitrogen fro ambient. Additional nitrogen thus comes from the ambient sour nother way is to deposit a thin oxy-nitride metal diffusion ba layer by using an oxygen/nitrogen precursor together wit precursors needed to deposit the nitrided metal diffusion ba . Not all oxy-nitrided materials however exhibit good adhesi perty.

Brief Summar t (22):
Cleaning the usion barrier, a method proposed by Charneski et al., in U.S. No. 6,... ,637 has a very limited success because it does not e the fundamental properties of the diffusion barrier surf Oxidizing the diffusion barrier, a method proposed by Nguyen et in U.S. Pat. No. ...3,144, is a proper method of improving th esion property. However, the improvement in adhesion is t by the introduction of a non-conductive oxide layer with t e of reactive oxygen species from a predominately oxygen plasm uyen et al.'s method solved the high contact resistance p m related to the non-conductive oxide layer by limiting the kness of this layer to less than 3 nm and relying on the tunn phenomenon to provide the conduction path.

Brief Summar t (23):
An improved d to improve adhesion is to form a multilayer: a nitrided met ffusion barrier layer and a thin oxy-nitride metal layer. The xy-nitride metal layer comprises the metal, nitrogen, a gen, ... the same oxy-nitride metal, the oxygen species bei s than the nitrogen species, that works best since

it is much m... onducti... than an oxide, and the adhesion property does not dif... ignifica...ly.

Brief Summa... (24):
Accordingly, ...thod to ...prove the adhesion property of copper to the nitrided ... diffu...on barri...s such as TiN, TiSiN, TaN, TaSiN, WN, W... by form...g a multilayered diffusion barrier structure i... ided. The provided multilayered diffusion barrier is deposite... n under...yer, and provides improved adhesion to a subsequently ...sited l...r. The method comprises the steps of: a) Depositing a ...rided me... diffusion barrier layer on the underlayer ... iffusion barrier deposition equipment, whereby this diffus... rrier l...r serves as a barrier between the underlayer ... subse...ently deposited layer; b) Forming a thin metal nitro... h oxy-...ride layer by oxy-nitriding the diffusion b... materi... surface, ...s oxy-nitride layer comprises t... al, nit...gen, and oxygen, the oxygen species being less than t... rogen sp...ies, whereby this layer serves to improve the ... sion of ... multilayered diffusion barrier structure t... subsequ...ly deposited layer.

Brief Summa... (25):
The two lay... itrided ...etal diffusi...n barrier layer and the oxy-nitride ... layer are formed i... sequence. The method includes th... ation o... these two layers in two separate locations: ... ocess c...bers in the same process equipment, or in two sepa... rocess ...ipment. The formation of the nitrided metal diffu... rrier w... occur in ...he first process chamber (of the two cha... quipment ...or in the ...irst process equipment. The substrate w... n be m...d to the s...ond process chamber (of the two chamber ...ent), ... to the sec...d process equipment for the oxy-nitridi... ess. T... oxy-nitridi...g process to form the oxy-nitride ... layer ...ll require an oxygen-contained ambient. In some asp... the i...ntion, an elevated temperature is also provided. T... ...nted t... erature ranges from 200.degree. C. to 1200.degree ... ith 30... 00.degree. ... being a typical temperature ... The ox... nitriding time ranges from a few seconds to several ... , depe...ing on many factors such as the optimizatio... ocess ...ow, the opt...mization of the desired level of adhesion ... ptimiz...ion of con...t resistance, etc.

Brief Summa... (27):
The method ... ides ... thickness of the oxy-nitride layer on the surface ... ided ... diffusi... barrier to be less than 5 nm. The 5 n... ...ie... tride layer offers adequate adhesion, a... ... much ...e overall thickness of the multilayere... ...n b... rier struc...e. Some applications, such as the ones ...ed ... conductor processing, require that the overall dif... ...rie... thickness ... be less than 50 nm, so a thin metal ... l... r is desirable, provided that adequate adhesion is ...

Brief Summa... (28):
The method ... ides ... techniques to deposit the nitrided metal diffu... er. ... evapora... n technique can deliver the nitrided me... ...en ... ...ting the source material. The sputter ... que ... deliver the nitrided metal diffusion